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(54) CERIUM-BASED POLISHING MATERIAL SLURRY AND METHOD FOR MANUFACTURING THE SAME

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a cerium-based polishing material slurry, and a method for manufacturing the same, high in polishing capability and in precision and not leaving grains on the polished surface.
SOLUTION: The slurry contains 95 wt.% or more of cerium-based polishing grains on total rare earth oxide (TREO) basis, the grains contain less than 3 wt.% of fluorine relative to TREO, and the grain diameter distribution determined by the laser diffraction method falls in a specified range. The slurry is further improved in adhesiveness when the fluorine rate is decreased to 0.005-0.5 wt.% relative to TREO.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to a cerium system abrasives slurry and a manufacturing method for the same. It is related with a cerium system abrasives slurry with little remains of the abrasives particles on the surface of an abrasives and adhesion after polish in detail.
[0002]
[Description of the Prior Art]The cerium system abrasives which contains cerium system particles as the main ingredients has extended the use quickly by the outstanding polish effect. In the present, it is used not only for the conventional glass polish use for optics but for the field of objects for electronic circuit manufacture, such as objects for magnetic recording media for glass polish, such as an object for liquid crystals for glass, and a hard

disk, and LSI.
[0003]This cerium system abrasives is generally manufactured by the following methods. That is, after slurring and carrying out the wet milling of the raw material and processing with mineral acid etc. if needed, fluoric acid, ammonium fluoride, etc. perform chemical preparation. And after filtering, drying and roasting the obtained slurry, grinding and a classification can be performed and the abrasives particles which have desired particle diameter can be obtained. As a raw material of this cerium system abrasives, rare earth raw materials, such as rare-earth carbonate, a hydroxylation rare earth, and an oxalic acid rare earth, or the oxidation rare earth raw material produced by calcinating these is used. Generally these rare earth raw materials are manufactured by removing some of rare earths (Nd, Pr, etc.), radioactive elements, etc. by chemical preparation from the bastnaesite system rare earth raw material or the cerium content rare earth raw material. Usually this cerium system abrasives is used by the slurry regime which carrier fluid, such as water, was made to distribute.
[0004]
[Problem(s) to be Solved by the Invention]By the way, demonstrating a high polish speed considers it as a premise, and a cerium system abrasives slurry is asked, and is further urged to be able to manufacture the abrasive face which has the outstanding mirror plane nature. This is the characteristic for which not only a cerium system but a general abrasives is asked. On the other hand, it not only excels in the polish characteristic, but the improvement of the other characteristics is called for.
[0005]As the new characteristic demanded from the cerium system abrasives slurry, one of them has adhesion over the abrasives surface of abrasives particles. In the field for which the cerium system abrasives is used, this relates to highly precise abrasive face formation being called for. That is, when the adhesion of the abrasives particles in an abrasives slurry is high, abrasives particles will remain on the abrasives surface after washing. And this remains abrasives particle causes a crack depending on subsequent handling, and will injure the accuracy of an abrasive face. And for example, if an abrasives is a substrate for hard disks, existence of such particles remaining will become a factor which worsens the smooth nature, when a magnetic body is vapor-deposited after polish.
[0006]On the other hand, the example of examination about an old abrasives or abrasives slurry has many examples about an improvement of the polish characteristic. However, to an adhesive improvement of the abrasives particles in an abrasives slurry, sufficient solution is not yet proposed, but there is nothing that aimed at coexistence with the polish characteristic and dispersibility.
[0007]This invention is made in view of the above-mentioned actual condition, and is a thing.
The purpose is excellent in polish accuracy, and it is providing the cerium system abrasives slurry which can maintain a highly precise mirror plane without generating generating of a crack after polish, without abrasives particles adhering to the abrasive face after polish.
The method that such a cerium system abrasives slurry can be manufactured is also provided.
[0008]
[Means for Solving the Problem]That this technical problem should be solved, this

invention persons resulted in the following considerations, as a result of examining first a cause by which the conventional cerium system abrasives particles adhere to an abrasives. In the conventional cerium system abrasives, fluoride is contained not less than (TREO standard) 3%. It is for reservation of polish power of a cerium system abrasives to make fluoride contain in a cerium system abrasives. Namely, although mechanical scouring by abrasives particles which consist of cerium oxide etc. is main in the conventional cerium system abrasives, In addition, it is for making fluoride form in the abrasives surface with fluoride to contain, also demonstrating simultaneously chemical scouring of promoting erosion of an abrasives, and raising polish power.
[0009]From such a reason, in the conventional cerium system abrasives, a fluorine component is made indispensable for polish power reservation, but on the other hand it is considered that fluoride becomes a factor which makes abrasives particles adhere to the abrasives surface after polish in the middle of the reactivity, therefore polish. In order for this invention persons to consider it as an adhesive low cerium system abrasives first paying attention to this point, It was thought that it became possible by reducing the reactivity of abrasives particles and a charge of an abrasives, and I thought that that (it will change into a state of being fluoride-free, thoroughly if possible) which reduces the amount of fluoride in a cerium system abrasives for that purpose was preferred.
[0010]In a grinder style of a cerium system abrasives as mentioned above on the other hand, A rate that a chemical operation by existence of fluoride occupies being also large, and only reducing the amount of fluoride to the conventional abrasives for adhesive reduction will also make polish capability of an abrasives slurry decline, and it will spoil the original function.
[0011]Then, this invention persons presupposed that this problem is solvable by controlling some characteristics of an abrasives, as a result of inquiring that it should consider as a cerium system abrasives slurry which has a high polish speed, even if it was in a state where fluoride was reduced. And it was considered as this invention for being regulating particle diameter and particle size distribution of abrasives particles to a prescribed range as the most effective characteristic.
[0012]Namely, the invention in this application a cerium system abrasives of 95 % of the weight or more of all the rare earth oxides (TREO) in an included cerium system abrasives slurry said cerium system abrasives, 10% of accumulation particle diameter [in / a fluorine content to TREO is less than 3 % of the weight, and / laser diffractometry] (D_{10}), 50% of accumulation particle diameter (D_{50}), 90% of accumulation particle diameter (D_{90}), and a diameter of grain of maximum size (D_{max}) are the cerium system abrasives slurries in following within the limits.
[0013]0.1 micrometer<=D $_{10}$<=0.6micrometer0.2 micrometer<=D $_{50}$<=1.0micrometer0.3 micrometer<=D $_{90}$<=1.5micrometer0.5 micrometer<=D $_{max}$<=5.0micrometer [0014]It is because, as for having made a fluorine content into less than 3 % of the weight, less than this figure needs to carry out at least here as fluorine concentration which an attachment phenomenon by fluoride of abrasives particles does not produce. And as for this fluorine content, if relation between performance as an abrasives and adhesion of abrasives particles is taken into consideration, it is preferred especially to consider it as 0.005 to 0.5% of the weight of a range. 0.005% was made into a lower limit of a fluorine content because a life of an abrasives became it short especially that the amount of cerium oxide is a freelancer with entire fluoride in 70% or less (TREO standard) when the amount of

cerium oxide in a cerium system abrasives is low.

[0015]And in this invention, cumulative size distribution of a maximum droplet size of abrasives particles and particle diameter is regulated in such a cerium system abrasives slurry of a low fluorine content. Having made a maximum droplet size (D_{max}) into the range of 0.5-5.0 micrometers here, When D_{max} is set to not less than 5.0 micrometers, while a crack will occur in an abrasive face and polish accuracy will be injured seriously, it is because it will be too fine and sufficient polish speed will not be obtained, if D_{max} shall be less than 0.5 micrometer. On the other hand, about particle size distribution, desirable particle size distribution is strictly specified by regulating particle diameter of abrasives particles in three points, 10% of accumulation particle diameter (D_{10}), 50% of accumulation particle diameter (D_{50}), and 90% of accumulation particle diameter (D_{90}). First, about 10% of accumulation particle diameter, having defined this range about these accumulation particle diameter has too fine abrasives particles, when this shall be less than 0.1 micrometer, and a problem of fluoride is because an adhesive problem arises independently, and is because it will become easy to generate a polish crack if it exceeds 0.6 micrometer. It is because it will become easy to generate a polish crack at less than 0.2 micrometer about 50% of accumulation particle diameter if polish power is insufficient, polish speed is inferior and it exceeds 1.0 micrometer. And while it is because abrasives particles in which polish speed is inferior and which upper-adhere increase at less than 0.3 micrometer about 90% of accumulation particle diameter, it is because it will become easy to generate a polish crack if it exceeds 1.5 micrometers. This range is limited further and it depends for it, and as a desirable range, it is 0.2 $<=D_{10}<=$0.4micrometer, 0.3 $<=D_{50}<=$0.6micrometer, and 0.5 $<=D_{90}<=$0.8micrometer, and is 0.8 $<=D_{max}<=$3.0micrometer.

[0016]These particle size distribution is defined on the basis of what is measured by laser diffractometry. Laser diffractometry is the technique of searching for particle size distribution from a diffraction pattern acquired by condensing the scattered light (forward scattering light) which makes carrier fluid distribute an abrasives, irradiates this with a laser beam, and is produced in this case. In this invention, it was based on laser diffractometry because this method method measured particle size distribution of an abrasives comparatively simply and promptly, and it is because it excels also in that accuracy of measurement.

[0017]By this invention, it is considered as a cerium system abrasives slurry without adhesion in an abrasive face as mentioned above, having polish speed sufficient [regulating a fluorine content to low concentration] by limiting particle diameter of abrasives particles, and particle size distribution in a prescribed range. And according to this invention person etc., if polish speed and accuracy of an abrasive face are taken into consideration, such performances are securable by regulating about the following two characteristics at a higher dimension.

[0018]The 1st characteristic for raising polish speed and accuracy of an abrasive face is a crystallite diameter and a crystal lattice constant of microcrystal which constitute abrasives particles. Abrasives particles are the aggregates of a single crystal and microcrystal is a single crystal of a rare earth oxide containing cerium oxide or cerium which constitutes this abrasives particle. And a crystallite diameter means a size of a plane direction (111) of this single crystal in this invention. On the other hand, a grating constant is a constant which specifies a size and shape of a unit cell which constitutes a

single crystal. In this invention, it is considered as a cerium system abrasives which has a path of this microcrystal, and a polish speed excellent in specifying a grating constant of a crystal which constitutes microcrystal and accuracy of an abrasive face. And it is preferred that a crystallite diameter is 10-50 nm, and a grating constant sets to 0.545-0.555 nm in this invention. A crystallite diameter is a size of a single crystal which constitutes abrasives particles, and while polish speed will become easy to generate a crack although it size-comes to come if a crystallite diameter becomes large, it is because opposite side polish speed will become low too much although generating of a crack is controlled if a crystallite diameter is small. About a grating constant, if a grating constant is large, a crystal can make polish speed high firmly, but a crack is made generated, if a grating constant is conversely small, a crystal will become soft and polish power will become low. A mentioned range is set up in consideration of the above. These crystallite diameters and a value of a crystal lattice constant are based on a value measured by powder X-ray diffraction analysis which is a general measuring method.

[0019]The 2nd characteristic for raising polish speed and accuracy of an abrasive face is the specific surface area of abrasives particles. And in this invention, the range of this specific surface area is set to 3-30-m^2/g. It is because it becomes easy to generate a polish crack in less than 3-m^2/g, and is because polish speed will become small if 30-m^2/g is exceeded. Specific surface area of this abrasives is based on a value measured by a BET adsorption method in dryness and a thing which dried an abrasives slurry at 105 ** in detail.

[0020]As for a ratio of cerium oxide to TERO, in a cerium system abrasives slurry which controlled a fluorine content explained above and particle size distribution and also a grating constant, and specific surface area, it is preferred that it is 30 % of the weight or more. It is because polish speed becomes extremely low at less than 30%. In order for polish speed to use polish power as an abrasives which can be held for a long period of time greatly especially, it is preferred to consider it as not less than 90%, but material cost will become high if it becomes not less than 99.99%.

[0021]About a cerium system abrasives slurry concerning this invention, that in which a thing of an anticaking agent and a dispersing agent which contains either at least included these both sides especially preferably is preferred. Here, when an anticaking agent makes carrier fluid distribute an abrasives abrasive grain, it is an additive agent which makes precipitate of an abrasives abrasive grain soft, and a dispersing agent means an additive agent which distributes an abrasives abrasive grain in carrier fluid. As this anticaking agent, silica, such as synthetic silica, colloidal silica, and fumed silica, Alginic acid or alginic acid derivatives, such as sodium alginate and propylene glycol alginate, A salt of aromatic-sulfonic-acid formalin condensate, such as beta-naphthalene sulfonic acid soda formalin condensate, Hydrogencarbonates, such as cellulose, such as calcium content compounds, such as calcium secondary phosphate, crystalline cellulose, and carboxymethylcellulose sodium, or a cellulosic and sodium bicarbonate, and ammonium acid carbonate, are mentioned. moreover -- as a dispersing agent -- condensed phosphoric acid, such as polystyrene sulfonate salts, such as sodium polystyrene sulfonate, pyrophosphoric acid, specific pyrophosphate, a specific tripolyphosphate, and hexaspecific metaphosphate, or condensed phosphate -- and, Polycarboxylic acid type high molecular compounds, such as a polyacrylic acid, polymer lane acid, and acrylic acid-maleic acid copolymer, polyacrylate, polymer lane acid chloride, and an acrylic

acid-maleic acid copolymer salt, are mentioned. Even if these additive agents are chosen by charge of an abrasives, and receive the usual glass material and an additive agent of a gap is used for them, they are satisfactory, but as for application of an additive agent which contains that of an alkaline metal and alkaline-earth metals to polish of a charge of semiconductor material, avoiding is preferred. For such materials, it is because existence of alkaline metals, such as sodium and calcium, and alkaline-earth metals is not preferred.

[0022]And an addition of these anticaking agents and a dispersing agent, receiving the cerium system abrasives particles 100g, when we decide to adjust according to specific surface area of an abrasives in a slurry and specific surface area of a cerium system abrasives is set to S (m^2/g) -- respectively -- S/100-S/5g -- adding is preferred. It is because a difference is not seen by the effect less than [S/100g] even if an effect as an additive agent is not demonstrated but it exceeds S/5g. for example, specific surface area of an abrasives -- the above -- when it is 3-30m^2/g which is a suitable range, it is preferred to carry out 0.03-6g (0.03 to 6 % of the weight) addition to the cerium system abrasives particles 100g. As for an addition of these anticaking agents and a dispersing agent, when adding independently, or when adding both sides, it is preferred to consider it as said range. When carrying out both addition of an anticaking agent and the dispersing agent, it is preferred for a ratio of an anticaking agent addition and a dispersing agent addition to be referred to as 1 / 20 - 20/1. Although it may be any under [before finish grinding mentioned later] finish grinding and after finish grinding, since an addition stage of these additive agents carries out [preferably] refining of the abrasives particle surface, it is preferred that it shall be before finish grinding. However, when passing a filter, since it will become easy to pass a filter if it adds before passing a filter, it is desirable.

[0023]It is preferred that it is 10 to 60 % of the weight at the time of manufacture, and if slurry concentration (content of abrasives particles) of a cerium system abrasives slurry concerning this invention is 20 to 60 % of the weight, it is still more preferred. It is because cost starts conveyance and storage at less than 10 % of the weight, and is because it is difficult to manufacture 60% of the weight or more of an abrasives slurry. And at the time of use, an abrasives slurry concerning this invention is diluted and used for proper concentration (0.1 to 30 % of the weight). Even if a cerium system abrasives slurry concerning this invention is high concentration, it excels in dispersibility, and also after prolonged storage, re dispersion by stirring by the usual agitator is possible, and the almost same particle size distribution as the time of manufacture is acquired. Even if sedimentation arises, a sedimentation cake is soft and re dispersion is easy for especially a thing that added an anticaking agent.

[0024]A cerium system abrasives slurry concerning this invention can be broadly applied to polish of a glass material and a semiconductor material, and is suitable for polish of a charge of liquid crystal material, and a substrate for magnetic recording media like a hard disk especially.

[0025]Next, a manufacturing method of an abrasives slurry concerning this invention is explained. A manufacturing method of an abrasives slurry concerning this invention improves a process to a roast process to a manufacturing method of a common cerium system abrasives, and also includes a process of slurring a roast article. Namely, a stock slurry chemically-modified [which manufacture of a cerium system abrasives mixes an abrasives raw material and carrier fluid, and it makes into stock slurry] degree, In order

to include a raw material grinding process which carries out the wet milling of this stock slurry, and a roast process of roasting an abrasives raw material after raw material grinding and to make this into an abrasives slurry, a process of slurring an abrasives raw material after roast is included. He is trying to become in this invention within the limits to which particle diameter of abrasives particles and its distribution were limited, reducing a fluorine content. It is contained also when slurrying is made in early stages in a wet-milling process besides in case a process of mixing carrier fluid and a raw material (roast article) with slurrying in this invention is independently. For example, when grinding by paying carrier fluid and a raw material (roast article) to a wet grinding mill of a batch type, both slurrying and wet milling are made.

[0026]Here, about reduction of fluorine concentration, it is possible by performing grinding and roast, without performing fluoridation processing currently performed by a manufacturing process of the conventional abrasives slurry about an abrasives raw material. since a fluorine component in a raw material volatilizes in a roast process, even if it performs fluoridation processing like before, it is considered as an abrasives slurry with low fluorine concentration applied to this invention by reducing an addition of fluoride -- things can be carried out. That is, when performing fluoridation processing, fluorine concentration of a final abrasives slurry can be made into less than 3% because fluorine concentration at the time of fluoridation processing considers it as 3% or less to TREO in a raw material.

[0027]And in this invention, particle diameter of abrasives particles and control of particle size distribution shall be performed in a grinding process explained below. Thus, although it is hard to grind an abrasives raw material after roast firmly and grinding after roast can also be adjusted about D_{50} of abrasives particles as a reason for thinking as important a grinding process in front of a roast process, It is because adjustment after roast is difficult about D_{90}/D_{10}, and it is necessary to make small D_{50} of a raw material before roast, and D_{90}/D_{10} in order to adjust D_{90}/D_{10}. That is, it is because it is preferred to set up grinding conditions before roast and it can also reduce grinding cost, in order to consider it as the range of a request of particle diameter of an abrasives.

[0028]A balancer product presupposes this grinding process that the main grinding is performed with a wet medium mill provided with tumbling media of $1.4x10^{-5}$ - $4.2x10^{-3}cm^3$ / individual. Although a spherical thing is usually used these tumbling media, when tumbling media make it a globular form, that diameter is set to 0.3-2.0 mm, but. In order for tumbling media of a major diameter to grind a raw material small enough, it can make D_{50} an appropriate range that tumbling media of this byway grind in this invention, even if time is taken too much, and it is inefficient and it ignores and grinds efficiency, but. It is because D_{90}/D_{10} becomes excessive and particle size distribution of a final abrasives cannot be carried out in an appropriate range. Then, this invention persons are making tumbling media into such a range, as a result of considering relation of final abrasives particle diameter, raw material particle diameter before roast, and tumbling media at the time of raw material grinding, Particle size distribution of raw material particles after grinding was made proper, and it found out that it could store in fitness within the limits which described above particle size distribution of a still more final abrasives. And as particle size distribution of raw material particles after grinding in this case, ten or less range has [D_{50}] 0.3-3.0 micrometers and D_{90}/D_{10} proper according to this invention person etc.

[0029]And in this invention, it is preferred to perform preliminary grinding explained below before the above-mentioned main grinding. It is because it is not efficient that tumbling media of a byway grind a raw material promptly and a long time is taken to consider it as an abrasives which has desired particle size distribution. D_{90}/D_{10} of raw material particles after the main grinding can be made small by performing the following preliminary grinding. And the following preliminary grinding is combined independently or suitably, and is performed before the main grinding.
[0030]A process which carries out wet milling as 1st preliminary grinding process with tumbling media of a 1.4 to 1000 times as many balancer product as tumbling media used by the main grinding process is mentioned. Thus, by performing beforehand wet milling using bigger tumbling media than the main grinding, a raw material can be ground with a sufficient degree and a grinding process by tumbling media of small size which is the main grinding can be performed efficiently. As an example of a suitable wet medium mill for this 1st preliminary grinding process, a wet ball mill, attritor, a vibration mill, etc. are mentioned.
[0031]As the 2nd contents of preliminary grinding, a raw material is immersed in solution (60 ** - 100 **). Thus, by heating a raw material in the state where you made it immersed into solution, a raw material collapses, and the particle diameter becomes fine and it becomes uniform [raw material particle diameter after collapse] further. Although this process differs from a general grinding process performed using a grinding apparatus, it is considered as preliminary grinding before the main grinding by this invention using this phenomenon. This preliminary grinding process is effective, when temporary quenching of rare-earth carbonate mentioned later or the rare-earth carbonate is carried out and ignition loss after 1-hour heating uses as a raw material what it is with 1 to 40% on a dry weight standard at 1000 **. And cooking temperature of this solution shall be 60 ** - 100 **. It is because grinding may not fully progress at less than 60 **, and is because it finds industrially that special devices, such as autoclave, are needed for heating at temperature over 100 ** etc. and it is disadvantageous it. [it] [it] A solution with which a water-soluble organic solvent of water (pure water, industrial water, tap water, etc.), for example, alcohol, acetone, etc., themselves was mixed is contained in solution in which a raw material is made immersed.
[0032]As a mode of this process, after making a raw material immersed into solution before heating, a mode which heats these, a mode which makes a raw material immersed into heated solution, a mode heated to a back pan made to immerse a raw material into heated solution, etc. can be considered, but it is good by all. As for solution, although the mixing ratio (wt. ratio) in particular of a raw material and solution is not limited, 0.5 time - 10 times of a raw material (when a raw material is a slurry, it is the formed element) are preferred. Even if it performs this preliminary grinding method after the main grinding process, it is effective.
[0033]And as the 3rd preliminary grinding, dry grinding is performed before the main grinding (wet milling). Since grinding D_{90}/D_{10} by medium mills, such as a dry type ball mill, becomes comparatively large about this dry grinding, It is carried out in a grinding means which particles are made to collide and is pulverized in a high-speed flow like a grinding means of a xenomorphic type, i.e., a grinding means pulverized by a shock, (a means to grind with a shock with pins, such as an impact mill and an atomizer, and a hammer), and a jet mill.

[0034]By this invention, the main grinding and proper preliminary grinding grind a raw material as mentioned above. And it can be considered as a cerium system abrasives concerning this invention by roasting a raw material ground by doing in this way. As for a raw material in which this main grinding and preliminary grinding were made, roasting, after filtering, drying and cracking is preferred.
[0035]Here, as for roast temperature in a roast process, it is preferred to consider it as 800-1100 **. It is because sintering of abrasives particles progresses too much, a crystallite diameter is excessive, and a grating constant will become large and will cause crack generating, if roast temperature exceeds 1100 **. It is because sintering of abrasives particles hardly advances, but a crystallite diameter becomes small, a grating constant becomes small and it becomes an abrasives with low polish power in roast below 800 **.
[0036]In order to control more strictly final particle size distribution of a cerium system abrasives, it is preferred to perform finishing grinding which carries out the wet milling of the raw material after roast with a wet medium mill provided with tumbling media of $3.0x10^{-6}$ - $5.3x10^{-4}cm^3$ / individual. A size of tumbling media in this case is specified because [of adjustment of particle size distribution]. while making D_{50} small by this -- D_{90}/D_{10} -- it can be made small. A slurry after this finish grinding becomes possible [using it as an abrasives slurry as it is], and it becomes unnecessary to perform a classification process thereby. Before this finishing grinding, extreme big particles can be eliminated by cracking a roast article, and finish grinding can be performed efficiently.
[0037]About a preliminary grinding process described above, the main grinding process, and a finish grinding process, if it is a batch type, in the case of a passage method, passing time shall be adjusted suitably, and grinding time etc. shall be performed. In the main grinding process and a finish grinding process, a planetary ball mill, a sand mill, a bead mill, etc. are mentioned as an example of a suitable wet medium mill.
[0038]It is also effective to slur a raw material after roast and to pass a filter with an aperture of 0.5-5 micrometers for this slurry. As for especially this filtering, it is preferred to be aimed at a slurry after performing the above-mentioned finish grinding. A slurry of a roast article may have too large particle size distribution, and it is because time of filtering will be taken in that case and also a yield of a product will get worse. And a filter of form like a cartridge filter as this filter can be applied, and a raw material may be passed one by one, combining a filter with which apertures differ two or more.
[0039]Considering cost of conveyance and storage, as for slurry concentration of a cerium system abrasives slurry manufactured by a method of this invention, it is desirable to make it as high as possible. Then, it may condense about an abrasives slurry manufactured. Although thickening which neglects an abrasives slurry, makes abrasives particles sediment as a condensation method, and extracts supernatant liquid is common, evaporation concentration by desiccation is also applicable. A cerium system abrasives slurry concerning this invention can be used as a cerium system abrasives of a solid state (powdered) by making it dry with a drying method which cannot cause dry condensation easily.
[0040]It is preferred whether ignition loss which carries out temporary quenching of rare-earth carbonate or the rare-earth carbonate, and is measured by heating at 1000 ** as a raw material of a cerium system abrasives concerning this invention for 1 hour is what it is with 1 to 40% on a dry weight standard, and *******. It is because one with these

preferred raw materials has few fluorine contents, and grinding of a raw material before roast is comparatively easy and sinters moderately by roast further.
[0041]Here, ignition loss (LOI (Loss On Ignition) may be called hereafter.) means a rate of weight loss at the time of igniting a subject. The range of LOI is made into 1 to 40% because less than 1% of raw material requires time for grinding and that whose grating constant a crystallite diameter is large and is too large also exists depending on the case. It is because it is difficult to make temporary quenching of the rare-earth carbonate more than LOI40% since LOI of rare-earth carbonate in the state where making 40% into upper limit does not have temporary quenching is about 40%.
[0042]After paying a raw material for abrasiveses dry enough at 105 ** to a crucible which measured weight beforehand as a measuring method of LOI and measuring the weight, After heating at 1000 ** in a furnace for 1 hour, it can cool radiationally under a drying atmosphere, and weight of a crucible after radiational cooling can be measured, and LOI can be calculated by following the following formula.
[0043]
[Equation 1]

$$B = (W_1 - W_2) / (W_1 - W_3) \times 100$$

(Weight (g) of the raw material for abrasiveses before B:ignition-loss (%) W_1:heating, the raw material for abrasiveses after weight (g) W_2:heating of a crucible, and a crucible, W_3: Weight of a crucible)
[0044]In the manufacturing process in the case of using as a raw material what carried out temporary quenching of this rare-earth carbonate, and made ignition loss 1 to 40%, as for this temporary-quenching temperature, although the process of carrying out temporary quenching of the rare-earth carbonate is needed, it is preferred to consider it as 400-800 **. It is for making a carbonic acid component emit moderately from rare-earth carbonate by carrying out temporary quenching at this temperature. That is, it is because change to an oxidation rare earth will be a perfect oxidation rare earth early and discharge of carbonic acid component sufficient below 400 ** and destruction of a coarse particle do not arise above 800 **.
[0045]A process suitable about the manufacturing method of the cerium system abrasives slurry concerning this invention explained above is shown in drawing 1.
[0046]
[Embodiment of the Invention]Hereafter, the suitable embodiment of this invention is described with a comparative example.
[0047]A 1st embodiment: The manufacturing process of the cerium system abrasives in this embodiment is the same in drawing 1. In this embodiment, a raw material is dried and cracked, preliminary grinding and after main-grinding. And after roasting this, finish grinding is performed, an additive agent is added to this, filtering is performed, and the abrasives slurry is manufactured. Although the abrasives slurry is directly manufactured in this embodiment, manufacture of the cerium system abrasives of a solid state (powdered) is possible by drying the slurry after filtering. Here, the fluorine concentration of a final abrasives slurry is adjusted with adjustment of the existence of fluoridation processing, and roast temperature.
[0048]<Raw material>: The raw material used here is rare-earth carbonate which has the following characteristics.

[0049]
TREO (dry weight standard) 67% cerium oxide concentration (TREO standard) 58% fluorine content (TREO standard) 0.16% moisture content (loss-on-drying method) 30% ignition loss (dry weight standard) 32% [0050]<The preliminary-grinding process of a raw material>: A steel ball 5 mm in diameter ($6.5x10^{-2}cm^3$ / individual) was put into the ball mill used as tumbling media, this 75 kg of raw materials and pure water 125L were ground for 4 hours, and it was considered as the preliminary grinding process.
[0051]<Main grinding process>: The raw material after preliminary grinding supplied stock slurry to the bead mill which used a zirconia ball 1.5 mm in diameter ($1.8x10^{-3}cm^3$ / individual) as tumbling media by about 2 L/min, ground it by the passage method, performed grinding 4 times (four paths), and considered this as main grinding.
[0052]Sample a proper quantity of stock slurry after the above main grinding, and it is put into sodium-hexametaphosphate solution 0.1% of the weight, the place (measuring device: -- SALD-2000A (made by Shimadzu).) which measured particle size distribution about that to which ultrasonic dispersion of this was carried out for 10 minutes In complex-index-of-refraction 2.20-0.10i, it was 0.647 micrometer in measurement and 10% of accumulation particle diameter (D_{10}), 1.583 micrometers in 50% of accumulation particle diameter (D_{50}), and 2.976 micrometers in 90% of accumulation particle diameter (D_{90}), and they were 8.38 micrometers of maximum droplet sizes (D_{max}).
[0053]<Fluoridation down stream processing> Next, divide the stock slurry after the main grinding into three equally, and it is supposed that the following roast process is performed without performing fluoridation processing about one, About the two [remaining], the ammonium fluoride solution was added and fluoridation processing was performed so that the fluorine concentration (TREO standard) in stock slurry might be 3% and 6%.
[0054]<Roast process> Filtered with the filter press, and heat for 40 hours, it was made to dry at 170 **, and three kinds of stock slurry more than : were further cracked with the roll crusher. And it roasted over the temperature which divides each into two equally and is shown below, respectively. The roast process was performed by holding to each temperature for 5 hours. And the raw material after roast was cracked with the sample mill.
[0055]
[Table 1]

フッ化処理	試料 No.	焙焼温度
フッ化処理なし	1	980℃
	2	1020℃
フッ素濃度 3 %	3	980℃
	4	920℃
フッ素濃度 6 %	比 1	980℃
	比 2	850℃

[0056]<Finishing grinding> : Mix 5.4 kg to the pure water 12.6L, and the raw material

after a crack is slurred, The planetary ball mill (number of rotations: 200 rpm of revolution, 300 rpm of rotation) which used a zirconia ball 0.3 mm in diameter ($1.4x10^{-5}cm^3$ / individual) as tumbling media for this performed grinding by the batch method for 2 hours. To the stock slurry after this grinding, it is an anticaking agent (crystalline cellulose (trade name "Avicel" (made by Asahi Chemical Co., Ltd.)) and a dispersing agent (sodium hexametaphosphate) were added 0.7% 0.4% to abrasives weight, respectively.) as an additive agent. And 7 micrometers in an aperture and a 3-micrometer cartridge filter were made to pass the slurry after additive agent addition one by one, and it was considered as the cerium system abrasives slurry.
[0057]After measuring particle size distribution about six kinds of cerium system abrasives slurries manufactured as mentioned above and making it dry at 105 **, a grating constant, a crystallite diameter, specific surface area, and fluorine concentration were measured. Measurement of specific surface area here was performed with the BET one point method for BET method and the gas flow method. Measurement of fluorine concentration was performed with heat hydrolysis separation and lantern alizarin complexone absorption photometry. About the value of fluorine concentration, the abrasives slurry is converted into the fluorine concentration value per TREO100 weight section from the fluorine concentration value about what was dried at 105 **, and the measured value of TREO in the dried abrasives slurry. These results are shown in Table 2.
[0058]
[Table 2]

No.	焙焼温度(℃)	F濃度(vs TREO%).	粒径分布(μm)				X線回折		比表面積(m^2/g)
			D_{10}	D_{50}	D_{90}	D_{max}	格子定数(nm)	結晶子径(nm)	
1	980	0.02	0.255	0.411	0.607	1.68	0.549	21	11.5
2	1020	0.014	0.302	0.488	0.723	1.68	0.551	28	9.3
3	980	2.5	0.283	0.536	0.772	2.51	0.547	37	9.1
4	920	2.7	0.241	0.507	0.690	2.05	0.548	33	11.2
比1	980	4.9	0.328	0.749	1.513	3.75	0.545	55	7.4
比2	850	5.3	0.217	0.520	0.783	2.51	0.547	40	11.2

[0059]And the glass material was ground about these abrasives slurries, and evaluation of the polish value and the abrasive face was carried out, and also the existence of the remains abrasives to an abrasive face was investigated by washing examination.
[0060]Polish of the glass material used the glass for flat panels as the abrasives with the high-speed grinder, and ground it using the polishing pad made from polyurethane. Polish conditions supplied the abrasives slurry at the rate of 25 ml/min, set the pressure to an abrasive face as 5.9kPa (0.06 kg/cm^2), and performed polish for 10 minutes for grinder revolving speed at 500 rpm. And evaluation of the polish value searched for the decrease of weight by polish by measuring the weight of the glass before and behind polish, and evaluated relatively the decrease of weight by the abrasives manufactured at the roast temperature of 920 ** as 100. Evaluation of the abrasive face irradiated the surface of the glass after polish with a 300,000 luxs halogen lamp, observed the glass surface with the

reflection method, carried out mark attachment with the size of a crack, and its number, and evaluated it by the demerit mark method from full marks.

[0061]Rinsing treatment which immerses the glass which is a thing to be ground in pure water for 10 minutes, and takes it in and out of pure water was performed 3 times, and the detergency examination was done by observing the dirt of an abrasive face by the dark field of an optical microscope after desiccation, after performing polish for 10 minutes in the above-mentioned polish test condition. And the relative comparison of the result of the detergency examination in each abrasives was carried out on the basis of the washing result in the comparative example 1, and each detergency was investigated.

[0062]Evaluation of the polish value by polish of this glass material and an abrasive face and an adhesion examination evaluation result are shown in Table 3. The photograph of the glass surface after sample No.1, 3, and the adhesion examination of the ratio 1 is shown in drawing 2 - drawing 4. In these photographs, the cerium system abrasives which remained to the abrasive face is reflected in the flake.

[0063]

[Table 3]

No.	焙焼温度 (℃)	F濃度 (vs TREO%).	研摩試験結果		
			研摩値	研摩面評価	付着試験
1	980	0. 02	100	100	◎
2	1020	0. 014	115	100	◎
3	980	2. 5	135	100	○
4	920	2. 7	121	100	○
比 1	980	4. 9	173	90	×
比 2	850	5. 3	131	96	×

◎ 残留（付着）なし（最終研摩用として極めて好適）

○ 僅かに残留（付着）有り（最終研摩用として好適）

× 付着あり（最終研摩用として使用不可）

[0064]From the result about the above this embodiment and comparative example, existence of stuck particles with it was checked. [an abrasives slurry inferior to the accuracy of the abrasive face of what has a high polish value and also concerning a comparative example, and] [remarkable in the abrasive face after polish] This is considered to be based on the fluoride contained in a comparative example. On the other hand, there was no such problem in the abrasives slurry concerning this embodiment, and there was no adhesive problem in it especially in 0.1% or less of fluorine concentration. He can understand this clearly also from drawing 2 - drawing 4, and, as for the abrasives slurry of sample No.1 and 3, it turns out that adhesion of abrasives particles is improved rather than the ratio 1.

[0065]A 2nd embodiment: Although this embodiment was the same contents as a 1st embodiment fundamentally, it changed each process of the main grinding process, the

roast process, and the finishing grinding process, and manufactured the abrasives slurry. The main grinding here performed 3 path grinding in the bead mill which used a zirconia ball 1.0 mm in diameter ($5.2x10^{-4}cm^3$ / individual) as tumbling media, and was taken as main grinding. When particle size distribution is measured about the raw material after this main grinding, the 10% of accumulation particle diameter (D_{10}) of 0.483 micrometer, It was 1.027 micrometers in 50% of accumulation particle diameter (D_{50}), and 2.138 micrometers in 90% of accumulation particle diameter (D_{90}), and they were 5.60 micrometers of maximum droplet sizes (D_{max}). And at the roast process, they could be 750 **, 950 **, 980 **, 1020 **, and 1050 **. In finishing grinding, grinding was performed for 2.5 hours or 3 hours in the planetary ball mill (number of rotations: 200 rpm of revolution, 300 rpm of rotation) which used a zirconia ball 0.65 mm in diameter ($1.4x10^{-4}cm^3$ / individual) as tumbling media. Other processes presupposed that it is the same as that of a 1st embodiment.

[0066]Particle size distribution, a grating constant, a crystallite diameter, and specific surface area were measured about five kinds of cerium system abrasiveses manufactured as mentioned above. This result is shown in Table 4.

[0067]

[Table 4]

No.	焙焼温度(℃)	仕上粉砕時間(h)	F濃度(vs TREO%)	粒径分布（μm）				X線回折		比表面積(m^2/g)
				D_{10}	D_{50}	D_{90}	D_{max}	格子定数(nm)	結晶子径(nm)	
5	750	2.5	0.075	0.258	0.437	0.673	2.51	0.542	8.8	16.7
6	950	3	0.025	0.228	0.347	0.546	1.37	0.548	17	14.6
7	980	3	0.019	0.259	0.423	0.620	1.68	0.549	22	11.7
8	1020	3	0.014	0.305	0.491	0.735	1.68	0.551	28	9.4
9	1050	3	0.008	0.330	0.576	0.789	2.05	0.553	35	7.3

[0068]And when the glass material was ground about these abrasiveses and the existence of evaluation of a polish value and an abrasive face and the remains abrasives to an abrasive face was investigated, the result of Table 5 was obtained.

[0069]

[Table 5]

No.	焙焼温度(℃)	仕上粉砕時間(h)	F濃度(vs TREO%)	研磨試験結果		
				研摩値	研摩面評価	付着試験
5	750	2.5	0.075	37	100	◎
6	950	3	0.025	89	100	◎
7	980	3	0.019	103	100	◎
8	1020	3	0.014	117	100	◎
9	1050	3	0.008	137	100	◎

◎　残留（付着）なし（最終研磨用として極めて好適）

[0070]A 3rd embodiment: Like a 2nd embodiment, this embodiment changed each

process of the main grinding process, the roast process, and the finishing grinding process, and manufactured the abrasives slurry. The main grinding here performed two pass grinding by the passage method of amount of liquid sending 2 L/min in the bead mill which used a zirconia ball 0.65 mm in diameter ($1.4x10^{-4}cm^3$ / individual) as tumbling media, and was taken as main grinding. When particle size distribution is measured about the raw material after this main grinding, the 10% of accumulation particle diameter (D_{10}) of 0.523 micrometer, It was 1.203 micrometers in 50% of accumulation particle diameter (D_{50}), and 2.335 micrometers in 90% of accumulation particle diameter (D_{90}), and they were 6.85 micrometers of maximum droplet sizes (D_{max}). And the roast process unified roast temperature with 980 **, and performed 2 - 8 path grinding by the passage method of amount of liquid sending 1 L/min in the bead mill which used a zirconia ball 0.4 mm in diameter ($3.4x10^{-5}cm^3$ / individual) as tumbling media for finishing grinding. Many things were adjusted to 3-7 in the aperture of the filter after finish grinding, and six kinds of abrasives slurries were manufactured.

[0071]Particle size distribution, a grating constant, a crystallite diameter, and specific surface area were measured about six kinds of cerium system abrasiveses manufactured as mentioned above. This result is shown in Table 6.

[0072]

[Table 6]

No	仕上パス数	フィルター	F濃度 (vs TREO%)	粒径分布（μm）				X線回折		比表面積 (m^2/g)
				D_{10}	D_{50}	D_{90}	D_{max}	格子定数 (nm)	結晶子径 (nm)	
10	2	10, 7	0. 020	0. 623	1. 225	2. 725	5. 60	0. 550	21	5. 8
11	3	10, 7, 5	0. 021	0. 435	0. 762	1. 225	3. 75	0. 549	21	7. 6
12	4	10, 5, 3	0. 020	0. 326	0. 518	0. 786	2. 05	0. 549	21	9. 4
13	5	10, 5, 3	0. 020	0. 248	0. 402	0. 597	1. 68	0. 549	21	11. 3
14	6	10, 5, 3	0. 020	0. 138	0. 267	0. 383	1. 37	0. 549	21	14. 4
15	8	10, 5, 3	0. 019	0. 075	0. 132	0. 285	1. 12	0. 548	20	19. 5

[0073]And the glass material was ground about these abrasiveses, the existence of evaluation of a polish value and an abrasive face and the remains abrasives to an abrasive face was investigated, and the result of Table 7 was obtained.

[0074]

[Table 7]

No.	仕上パス数	フィルター	F濃度(vs TREO%)	研摩試験結果		
				研摩値	研摩面評価	付着試験
10	2	10, 7	0.020	178	95	◎
11	3	10, 7, 5	0.021	142	98	◎
12	4	10, 5, 3	0.020	115	100	◎
13	5	10, 5, 3	0.020	99	100	◎
14	6	10, 5, 3	0.020	72	100	○
15	8	10, 5, 3	0.019	33	100	△

◎ 残留（付着）なし（最終研摩用として極めて好適）
○ 僅かに残留（付着）有り（最終研摩用として好適）
△ 中程度の残留（付着）有り（最終研摩用として使用可能ではあるが洗浄が必要）
× 付着あり（最終研摩用として使用不可）

[0075]A 4th embodiment: In this embodiment, it carried out by having changed various the methods of the preliminary grinding process, the main grinding, roast, etc. carried out these, and the abrasives slurry was manufactured. The process which does not perform preliminary grinding for comparison was also carried out. The method of preliminary grinding is as follows.
[0076](Wet milling) A steel ball 5 mm in diameter ($6.5x10^{-2}cm^3$ / individual) was put into the ball mill used as tumbling media, 25 kg of raw materials and the pure water 40L were ground for 4 hours, and it was considered as the preliminary grinding process.
(Immersion heating grinding) 25 kg of raw materials were immersed in the water 40L, and this was heated at 80 ** and neglected for 20 minutes.
(Dry grinding) The jet mill ground 25 kg of raw materials after 4-hour desiccation at 150 **.
[0077]The raw material after the above preliminary grinding performed two pass grinding by the passage method of amount of liquid sending 2 L/min in the bead mill which used a zirconia ball 0.65 mm in diameter ($1.4x10^{-4}cm^3$ / individual) as tumbling media, and considered it as main grinding. However, when he had no preliminary grinding, since D_{50} became large too much in a two pass, 4 path grinding was performed only in this case. When particle size distribution was measured about the raw material after this main grinding, the following particle size distribution was acquired.
[0078]
[Table 8]

No.	予備粉砕方法	粒径分布（μm）			
		D_{10}	D_{50}	D_{90}	D_{max}
16	湿式粉砕	0. 523	1. 203	2. 335	6. 85
17	浸漬加熱粉砕	0. 532	1. 137	2. 149	5. 60
18	乾式粉砕	0. 374	1. 226	2. 957	10. 25
19	なし	0. 328	1. 277	3. 894	18. 73

[0079]And the raw material after these main grinding was unified with 980 **, and roasted roast temperature, and the passage method of amount of liquid sending 1 L/min performed 5 path grinding in the bead mill which used a zirconia ball 0.4 mm in diameter ($3.4x10^{-5}cm^3$ / individual) as tumbling media as finishing grinding. After finish grinding, 10 and a 5 or 3-micrometer filter were passed, and the abrasives slurry was manufactured.
[0080]Particle size distribution, a grating constant, a crystallite diameter, and specific surface area were measured about four kinds of cerium system abrasiveses manufactured as mentioned above. This result is shown in Table 9.
[0081]
[Table 9]

No.	予備粉砕方法-	F濃度 (vs TREO%)	粒径分布（μm）				X線回折		比表面積 (m^2/g)
			D_{10}	D_{50}	D_{90}	D_{max}	格子定数 (nm)	結晶子径 (nm)	
16	湿式粉砕	0. 020	0. 248	0. 402	0. 597	1. 68	0. 549	21	11. 3
17	浸漬加熱粉砕	0. 019	0. 242	0. 379	0. 553	1. 37	0. 549	21	12. 3
18	乾式粉砕	0. 021	0. 192	0. 411	0. 732	2. 05	0. 549	21	11. 0
19	なし	0. 020	0. 183	0. 425	0. 937	4. 58	0. 549	21	10. 7

[0082]And the glass material was ground about these abrasiveses, the existence of evaluation of a polish value and an abrasive face and the remains abrasives to an abrasive face was investigated, and the result of Table 10 was obtained.
[0083]
[Table 10]

No	仕上パス数	F濃度 (vs TREO %)	研摩試験結果		
			研摩値	研摩面評価	付着試験
16	湿式粉砕	0.020	99	100	◎
17	浸漬加熱粉砕	0.019	107	100	◎
18	乾式粉砕	0.021	88	100	○
19	なし	0.020	83	99	○

◎ 残留（付着）なし

○ 僅かに残留（付着）有り

[0084]It was checked that a good result is obtained from the result of this 4th embodiment even if it changes the method of preliminary grinding.

[0085]An abrasives is manufactured so that the specific surface area of an abrasives may be set to 7-m^2/g according to the same manufacturing process as 5th embodiment:, next sample No.9 of a 2nd embodiment, The addition concentration of the additive agent was changed in this process, the abrasives slurry was manufactured, and, in addition to the polish examination and the washing examination, the sedimentation test and the sedimentation cake hardness test were done about these. Crystalline cellulose and a sodium hexametaphosphate were used for the additive agent here. The addition was made into 0 to 1.5% to abrasives weight according to the specific surface area of the obtained abrasives.

[0086]After a sedimentation test dilutes each abrasives slurry so that abrasives concentration may be 5 % of the weight, it is agitated enough here, Sedimentation nature is investigated from supplying this abrasives slurry to a 2000-ml measuring cylinder, and carrying out graduation reading of the interface of the suspension phase in an abrasives slurry, and a dispersion medium phase (pure water). And the evaluation value of this sedimentation test measures the capacity of a suspension phase, when it settles and passes for 60 minutes after supplying an abrasives slurry to a measuring cylinder, and it obtains it by computing the rate (%) over the total abrasives slurry capacity (2000 ml) of that measured suspension compatible quantity.

[0087]A sedimentation cake hardness test supplies each abrasives slurry in a predetermined container, by settling for 48 hours, it produces a precipitate of an abrasives abrasive grain, forms a sedimentation cake, and investigates the hardness of the sedimentation cake. Specifically the sedimentation cake was formed, operation which makes a container reverse and understands a cake was carried out once at 2 seconds, and it went this to 100 times.

[0088]The result of this example is shown in Table 11. The content of crystalline cellulose and a sodium hexametaphosphate is divided length and horizontally, respectively, and is shown in Table 11, and the result of having done the polish examination, the sedimentation cake hardness test, the detergency examination, and the sedimentation test, respectively is shown about the abrasives in each content. a sedimentation cake should understand a sedimentation cake hardness test result thoroughly -- the operation frequency to ** is estimating.

[0089]
[Table 11]

			結晶セルロース（wt%）					
	添加量		0	0.03	0.10	0.50	1.2	1.5
ヘキサメタ燐酸ソーダ (wt%)	0	A	103	101	98	87	84	80
		B	△	○	◎	◎	◎	◎
		C	◎	◎	◎	◎	◎	◎
		D	31	45	50	56	47	39
	0.03	A	105	105	105	102	95	85
		B	△	○	◎	◎	◎	◎
		C	◎	◎	◎	◎	◎	◎
		D	45	53	59	69	53	49
	0.10	A	106	107	113	126	117	92
		B	△	○	○	◎	◎	◎
		C	○	◎	◎	◎	◎	◎
		D	59	70	98	99	98	60
	0.50	A	109	108	117	137	124	96
		B	×	△	○	◎	◎	◎
		C	○	○	◎	◎	◎	◎
		D	71	75	99	100	99	63
	1.2	A	111	113	120	139	128	100
		B	×	△	○	◎	◎	◎
		C	○	○	○	◎	◎	◎
		D	70	73	98	99	98	59
	1.5	A	114	117	124	140	129	102
		B	×	△	○	○	◎	◎
		C	△	○	○	○	◎	◎
		D	58	60	66	71	63	53

A段；研摩値（第１実施形態の試料１を100とする）

B段；沈降ケーキ硬さ

◎　極めて良好（５回）　　○　良好（２０回）

△　中程度（１００回）　　×　不良（１００回以上）

C段；洗浄性

◎　残留（付着）なし　　○　僅かに残留（付着）有り

△　中程度の残留（付着）有り　　×　付着あり

D段；研摩材スラリー作製後６０分経過時の懸濁相容量割合（％）

[0090]An abrasives is manufactured so that the specific surface area of an abrasives may be set to 11-m^2/g according to the same manufacturing process as 6th embodiment:, next sample No.13 of a 3rd embodiment, The addition concentration of the additive agent was changed in this process, the abrasives slurry was manufactured, and, in addition to the polish examination and the washing examination, the sedimentation test and the sedimentation cake hardness test were done about these. Crystalline cellulose and a sodium hexametaphosphate were used for the additive agent here. The addition was made into 0 to 2.5% to abrasives weight according to the specific surface area of the obtained abrasives. This result is shown in Table 12.
[0091]
[Table 12]

ヘキサメタ燐酸ソーダ (wt%) 添加量		結晶セルロース (wt%) 0	0.08	0.13	0.80	2.0	2.5
0	A	68	68	64	58	53	50
	B	△	○	◎	◎	◎	◎
	C	◎	◎	◎	◎	◎	◎
	D	39	48	53	59	52	43
0.08	A	71	70	67	65	60	58
	B	×	○	◎	◎	◎	◎
	C	◎	◎	◎	◎	◎	◎
	D	51	57	63	71	56	53
0.13	A	73	73	80	95	83	65
	B	×	○	○	◎	◎	◎
	C	○	◎	◎	◎	◎	◎
	D	63	74	98	100	98	65
0.80	A	76	77	89	102	94	72
	B	×	△	○	◎	◎	◎
	C	○	○	◎	◎	◎	◎
	D	76	78	99	100	99	70
2.0	A	79	80	91	105	98	74
	B	×	△	○	◎	◎	◎
	C	○	○	○	◎	◎	◎
	D	72	75	99	99	98	61
2.5	A	82	84	93	106	99	74
	B	×	△	○	○	◎	◎
	C	△	○	○	○	◎	◎
	D	63	66	70	73	65	57

A段；研摩値（第１実施形態の試料１を 100 とする）

B段；沈降ケーキ硬さ

◎　極めて良好（５回）　○　良好（２０回）

△　中程度（１００回）　×　不良（１００回以上）

C段；洗浄性

◎　残留（付着）なし　○　僅かに残留（付着）有り

△　中程度の残留（付着）有り　×　付着あり

D段；研摩材スラリー作製後６０分経過時の懸濁相容量割合（%）

[0092]A 7th embodiment : An abrasives is manufactured so that the specific surface area of an abrasives may be set to 17-m^2/g according to the still more nearly same manufacturing process as sample No.5 of a 2nd embodiment, The addition concentration of the additive agent was changed in this process, the abrasives slurry was manufactured, and, in addition to the polish examination and the washing examination, the sedimentation test and the sedimentation cake hardness test were done about these. Crystalline cellulose and a sodium hexametaphosphate were used for the additive agent here. The addition was made into 0 to 4.0% to abrasives weight according to the specific surface area of the obtained abrasives. This result is shown in Table 13.

[0093]

[Table 13]

	添加量		結晶セルロース（wt%） 0	0.13	0.20	1.1	3.0	4.0
ヘキサメタ燐酸ソーダ (wt%)	0	A	55	54	51	46	42	40
		B	△	○	◎	◎	◎	◎
		C	◎	◎	◎	◎	◎	◎
		D	51	55	59	62	53	49
	0.13	A	58	58	57	55	53	48
		B	×	○	◎	◎	◎	◎
		C	◎	◎	◎	◎	◎	◎
		D	62	67	71	75	61	57
	0.20	A	60	62	75	82	77	60
		B	×	○	○	◎	◎	◎
		C	○	◎	◎	◎	◎	◎
		D	70	75	99	100	98	69
	1.1	A	62	63	80	85	82	63
		B	×	△	○	◎	◎	◎
		C	○	○	◎	◎	◎	◎
		D	78	79	99	99	98	65
	3.0	A	64	64	82	88	85	67
		B	×	△	○	◎	◎	◎
		C	○	○	○	◎	◎	◎
		D	75	77	99	99	98	65
	4.0	A	65	66	83	88	86	69
		B	×	×	○	○	◎	◎
		C	△	○	○	○	◎	◎
		D	68	71	76	79	69	61

A段；研摩値（第１実施形態の試料１を100とする）

B段；沈降ケーキ硬さ

◎ 極めて良好（５回）　○ 良好（２０回）

△ 中程度（１００回）　× 不良（１００回以上）

C段；洗浄性

◎ 残留（付着）なし　○ 僅かに残留（付着）有り

△ 中程度の残留（付着）有り　× 付着あり

D段；研摩材スラリー作製後６０分経過時の懸濁相容量割合（％）

[0094]It was checked that the ranges of the addition concentration of the additive agent which can aim at coexistence of the polish characteristic and adhesion (field enclosed with the thick line of each table) differ by each embodiment, i.e., the specific surface area of an abrasives, so that these [5th] - a 7th embodiment might show. And the appropriate range of this anticaking agent and a dispersing agent addition, In a 5th embodiment (specific surface area about $7m^2/g$), it was 0.1 to 1.2 % of the weight, was 0.13 to 2.0 % of the weight in a 6th embodiment (specific surface area about $11m^2/g$), and was 0.2 to 3.0 % of the weight in a 7th embodiment (specific surface area about $17m^2/g$). These additions have all S / within the limits of 100 - S/5, when specific surface area of a cerium system abrasives is set to S (m^2/g).

[0095]An 8th embodiment: Finally, as shown in Table 15, it evaluated by changing various combination of an anticaking agent and a dispersing agent. About evaluation, the same polish examination as the above-mentioned embodiment, the sedimentation cake

hardness test, the detergency examination, and the sedimentation test were done. What was manufactured like the abrasives slurry of 1st embodiment sample No.1 was used for the abrasives slurry used for evaluation. The result is shown according to Table 14.
[0096]
[Table 14]

固化防止剤（0.3wt%）	分散剤（0.3wt%）	A	B	C	D
ｱﾙｷﾞﾝ酸ﾌﾟﾛﾋﾟﾚﾝｸﾞﾘｺｰﾙｴｰﾃﾙ	ﾎﾟﾘｱｸﾘﾙ酸ﾅﾄﾘｳﾑ	100	◎	◎	100
ｱﾙｷﾞﾝ酸ﾌﾟﾛﾋﾟﾚﾝｸﾞﾘｺｰﾙｴｰﾃﾙ	ﾍｷｻﾒﾀ燐酸ﾅﾄﾘｳﾑ	99	◎	◎	100
ｱﾙｷﾞﾝ酸ﾌﾟﾛﾋﾟﾚﾝｸﾞﾘｺｰﾙｴｰﾃﾙ	ﾎﾟﾘｽﾁﾚﾝｽﾙﾎﾝ酸ﾅﾄﾘｳﾑ	98	◎	◎	100
結晶ｾﾙﾛｰｽ+ｶﾙﾎﾞｷｼﾒﾁﾙｾﾙﾛｰｽ	ﾎﾟﾘｱｸﾘﾙ酸ﾅﾄﾘｳﾑ	100	◎	◎	100
結晶ｾﾙﾛｰｽ+ｶﾙﾎﾞｷｼﾒﾁﾙｾﾙﾛｰｽ	ﾍｷｻﾒﾀ燐酸ﾅﾄﾘｳﾑ	105	◎	◎	100
結晶ｾﾙﾛｰｽ+ｶﾙﾎﾞｷｼﾒﾁﾙｾﾙﾛｰｽ	ﾎﾟﾘｽﾁﾚﾝｽﾙﾎﾝ酸ﾅﾄﾘｳﾑ	99	◎	◎	100
合成シリカ	ﾎﾟﾘｱｸﾘﾙ酸ﾅﾄﾘｳﾑ	97	◎	◎	100
第２燐酸カルシウム	ﾎﾟﾘｱｸﾘﾙ酸ﾅﾄﾘｳﾑ	100	◎	◎	100
βﾅﾌﾀﾚﾝｽﾙﾎﾝ酸ﾅﾄﾘｳﾑﾎﾙﾏﾘﾝ縮合物	ﾎﾟﾘｱｸﾘﾙ酸ﾅﾄﾘｳﾑ	100	◎	◎	100

A段；研摩値（第１実施形態の試料１を100とする）
B段；沈降ケーキ硬さ
◎　極めて良好（５回）
C段；洗浄性
◎　残留（付着）なし
D段；研摩材スラリー作製後６０分経過時の懸濁相容量割合（％）

[0097]As shown in Table 14, it became clear that the abrasives characteristic of these nine kinds of combination was almost equivalent to the characteristic data of the abrasives containing an equivalent amount of crystalline cellulose and sodium hexametaphosphates of a 1st embodiment.
[0098]
[Effect of the Invention]As explained above, the cerium system abrasives concerning this invention is adjusting the size of the tumbling media at the time of grinding, adjusts the particle size distribution of abrasives particles, and performs suitably implementation of preliminary grinding, adjustment of roast temperature, and implementation of finish grinding further. And according to the cerium system abrasives concerning this invention, it excels in polish power and polish accuracy, and a highly precise mirror plane can be maintained, without generating generating of a crack after polish, without abrasives particles adhering to the abrasive face after polish.

CLAIMS

[Claim(s)]

[Claim 1]In an included cerium system abrasives slurry, cerium system abrasives particles of 95 % of the weight or more of all the rare earth oxides (TREO) said cerium system abrasives particle, A cerium system abrasives slurry whose fluorine content to TREO is less than 3 % of the weight and which has 10% of accumulation particle diameter (D_{10}) in laser diffractometry, 50% of accumulation particle diameter (D_{50}), 90% of accumulation particle diameter (D_{90}), and a diameter of grain of maximum size (D_{max}) in following within the limits.

0.1 micrometer<=D $_{10}$<=0.6micrometer0.2 micrometer<=D $_{50}$<=1.0micrometer0.3 micrometer<=D $_{90}$<=1.5micrometer0.5 micrometer<=D $_{max}$<=5.0micrometer [Claim 2]The cerium system abrasives slurry according to claim 1 which has a fluorine content of a cerium system abrasives in 0.005 to 0.5% of the weight of a range to TREO.

[Claim 3]The cerium system abrasives slurry according to claim 1 or 2 whose crystallite diameter which constitutes abrasives particles is 10-50 nm and whose grating constant measured by powder X-ray diffractometry is 0.545-0.555 nm.

[Claim 4]The cerium system abrasives slurry according to claim 1 to 3 whose specific surface area measured by a BET adsorption method in dryness is 3-30m^2/g.

[Claim 5]The cerium system abrasives slurry according to claim 1 to 4 whose ratio of cerium oxide to TERO is 30 % of the weight or more.

[Claim 6]The cerium system abrasives slurry of an anticaking agent and a dispersing agent according to claim 1 to 5 which contains either at least.

[Claim 7]The cerium system abrasives slurry according to claim 1 to 6 which are S / 100 - S/5 (g) when content to the cerium system abrasives particles 100g of an anticaking agent and a dispersing agent sets specific surface area of a cerium system abrasives slurry of dryness to S (m^2/g).

[Claim 8]In a manufacturing method of a cerium system abrasives slurry characterized by comprising the following, a raw material grinding process, A manufacturing method of cerium system abrasives particles which consist of the main grinding process pulverized with a wet medium mill provided with tumbling media of 1.4×10^{-5} - $4.2\times10^{-3}cm^3$ / individual by a balancer product.

A raw material grinding process which carries out the wet milling of the stock slurry containing an abrasives raw material.

A process of roasting an abrasives raw material after raw material grinding.

[Claim 9]A manufacturing method of the cerium system abrasives particles according to claim 8 including a process of carrying out the wet milling of the raw material grinding process with tumbling media of a 1.4 to 1000 times as many balancer product as tumbling media used by the main grinding process before the main grinding process.

[Claim 10]A manufacturing method of the cerium system abrasives particle according to claim 8 or 9 with which a raw material grinding process includes a process to which a front stirrup of the main grinding process immerses a raw material in solution (60 ** - 100 **) behind.

[Claim 11]A manufacturing method of the cerium system abrasives particles according to claim 8 to 10 including a process to which a raw material grinding process grinds a raw material by dry type before the main grinding process.

[Claim 12]A manufacturing method of the cerium system abrasives particles according to

claim 8 to 11 over which a roast process roasts roast temperature as 800-1100 **.
[Claim 13]A manufacturing method of the cerium system abrasives particle according to claim 8 to 12 which perform this finishing grinding process including a finishing grinding process which carries out the wet milling of the raw material after roast with a wet medium mill provided with tumbling media of 3.0×10^{-6} - $5.3 \times 10^{-4} cm^3$ / individual.
[Claim 14]A manufacturing method of the cerium system abrasives particles according to claim 8 to 13 including a process which passes a filter with an aperture of 0.5-5 micrometers for a raw material after roast.
[Claim 15]A manufacturing method of the cerium system abrasives particles according to claim 8 to 14 which ignition loss which carries out temporary quenching of rare-earth carbonate or the rare-earth carbonate, and is measured by heating at 1000 ** as an abrasives raw material for 1 hour uses on a dry weight standard in what it is with 1 to 40%, and ********.